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(54) METHOD FOR PURIFYING VEGETABLE WAX

(57)Abstract:

PURPOSE: To purify the subject wax useful for foods, etc., by melting crude wax extracted from a plant, reacting the melted crude wax with a specific metal compound, adding a polar solvent thereto and carrying out deacidification and decoloring in simple operation reduced in load of cost.

CONSTITUTION: (A) Crude wax extracted from a plant is melted and a fatty acid isolated from the component A is made to react with (B) a divalent metal compound and (C) a polar solvent is added to the reactional product and the mixture is heated to 75-100°C and the wax component is extracted with the component C to purify the objective wax. Furthermore, as the component A, either one of carnauba wax, candelilla wax, rice wax and sugarcane wax is preferably used and as the component B, an oxide or a carbonate of an alkaline earth metal is preferably used and as the component C, a lower alcohol is preferably used.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] With respect to the purification method of the rough wax extracted from vegetation, this invention removes the pitch and free fatty acid in a vegetable rough wax, and relates to the technique of raising the quality of this wax.
[0002]
[Description of the Prior Art] There were carnauba wax, a candelilla wax, a rice ****** wax, a ********** wax, etc. as wax extracted from vegetation, and refining processing of depigmentation, deodorization, etc. has been performed in it in commercial production. Since these are ******s, they contain many ester, a hydrocarbon, long chain fatty acids of disengagement, long chain alcohol of disengagement, pitches, etc. Since especially a long chain fatty acid and a pitch cause various problems in [unlike other components, physical behavior's extending the intended-use domain of such a wax], although it is necessary to remove them in this, refining processing which changes component composition of a wax does not have an example industrially.
[0003] For example, carnauba wax is a vegetable wax obtained from the sheet of the vegetation of the Palmae called Copernicia cerifera Martius, and the melting point is as high as about 80-86 degrees C, and is excellent in the emulsification property including hydroxyl acid ester. Therefore, there is much intended use, such as finishing of cosmetics, a lustering agent, the ink for carbon, and leather, a precision casting, the drug, a polish agent, and coating. Usually, the other components of that to which composition differs and is called No. 1 wax by the site of the sheet of the palm from which carnauba wax is obtained are the alcohol of disengagement and a fatty acid, and about 3 - 4% of a pitch including a part for 80 - 85% of ester. However, the content for ester falls and a color tone also deteriorates as the quality of a wax deteriorates.
[0004] Especially a pitch increases to about 20 - 40% with a No. 2 wax and a No. 3 wax, and a blinding is produced at the time of handling, or it has troubles, like the solubility over ethanol is bad, and the elimination technique is examined by JP,2-279794,A. Moreover, the fatty acid of disengagement produces a phenomenon (bleeding) which wipes powder on the front face of a wax, and this phenomenon causes a quality degradation of various products and deterioration of the goods value of an exterior. The acid number of a wax produces this bleeding notably or more by about ten. Conventionally, the technique of making alkali soap in the esterifying method (JP,2-115300,A) or the organic solvent, and rinsing as a method of deoxidizing solid fat, etc. is learned. For example, although a rice ****** wax carries out the cooling precipitation of the rice ****** oil and it is obtained, it is carrying out alkali deoxidation of the hexane solution of rough rice ****** oil, and the deoxidation of a rice ****** wax is also performed simultaneously.
[0005]
[Problem(s) to be Solved by the Invention] However, although the above-mentioned esterifying method is simple as operation information, since it becomes synthetic compounds, it is hard to use it for a food-grade way. Moreover, although the organic acid is used for tinction prevention, there is an inclination which carries out a heat tinting conversely, and there is a problem also in respect of a color tone. Moreover, it is difficult for the technique of using as alkali soap and rinsing for a solution to emulsify in actual operation and to separate this soap from a wax. Therefore, the purpose of this invention is easy to operate it, and it is in offering the technique of performing simultaneously deoxidation of the rough wax extracted from vegetation, without causing the quality degradation by tinction, an improvement of a color tone, and elimination of a pitch.
[0006]
[Means for Solving the Problem] the efficient deoxidation is possible by removing the metallic soap to generate with a pitch using a polar solvent, after this invention persons make the fatty acid and divalent metallic compounds of the disengagement in the rough wax extracted from vegetation react zealously as a result of a research, in order to solve the

above-mentioned technical probrem -- the depigmentation effect also found out a certain thing collectively
[0007] That is, this invention is the purification method of the vegetable wax characterized by dissolving the rough wax extracted from vegetation, making divalent metallic compounds act on the free fatty acid in the aforementioned wax, adding a polar solvent to this, heating at 75-100 degrees C, and extracting a wax component by the polar solvent. Like sodium soap, 1 ********** has the good solubility to a polar solvent, and it does not turn [**********] to a separation with a wax. Moreover, the metallic soap more than trivalence, such as an aluminum soap, gels a polar solvent and is unsuitable. As a divalent metal, alkaline earth metal is suitable and especially calcium and/or magnesium are desirable. That is, in order to generate a metallic soap, a vegetable wax is dissolved and the end of a metal, an oxide, a hydroxide, or a carbonate of calcium and/or magnesium etc. is made to act. You may use these as independent or mixture. An oxide and a carbonate are desirable especially. One to 20 times of the amount of theory computed from the acid number of the vegetable wax which should be processed are suitable for the addition. Moreover, in order to promote a soap-ized reaction, coexistence of water, a dibasic acid, a hydrogen peroxide, a surfactant, polyhydric alcohol, an aluminosilicate, alkali-metal ion, etc. is effective.
[0008] In addition, once neutralizing the free fatty acid in a vegetable wax with alkali-metal hydroxides, such as sodium, there is a method of making it react in the chloride of alkaline earth metal or a sulfation object, and the aqueous solution, and obtaining a desired metallic soap. In the case of this technique, in order to raise the solubility of the wax to a polar solvent, in advance of extract operation, it is necessary to dehydrate a reaction mixture enough. Next, a polar solvent is added to the vegetable wax containing the divalent metallic soap obtained by the aforementioned operation, and a wax is melted at 75 degrees C - 100 degrees C. At this time, the unreacted metal or unreacted metallic compounds with an insoluble pitch and a divalent metallic soap superfluous as paste-like precipitate precipitates at the base of a solvent as a solid-state. After putting precipitate and supernatant liquor and dissociating, heating to such temperature, supernatant liquor is obtained by operation of a decantation etc., and if a solvent is removed by distillation, crystallization, or filtration processing, the target refining vegetable wax will be further obtained in this. As for the obtained refining vegetable wax, depigmentation is also carried out with the reduction of the acid number. Although lower alcohols, such as a methanol, ethanol, a butanol, propanol, and an isopropanol, an acetone, a methyl ethyl ketone, etc. are raised as a polar solvent used by this invention, the boiling point or a soluble point to ethanol or an isopropanol is desirable. Although an example is shown below, these do not limit the embodiment of this invention.
[0009]
[Example]
20g of carnauba wax the articles of No. 3 whose example 1 acid number is 15 was dissolved at 80 degrees C. 1g of magnesium oxides was added and it stirred for 30 minutes at 80 degrees C under reduced pressure. 100ml of ethanol was added to this, and it flowed back for 30 minutes. Furthermore, for 15 minutes and after putting at 75 degrees C, precipitation was removed by the decantation, ethanol distilling off of the supernatant was carried out, and it was condensed. This ethanol processing was repeated 3 times, and was performed, and 13g of the refining carnauba wax made into the purpose was obtained. The acid number of this refining wax is 7, and was decolorized compared with the raw material. Moreover, the pitch was a minute amount as a result of TLC (thin-layer chromatography) analysis.
[0010] 20g of carnauba wax the articles of No. 1 whose example 2 acid number is 3 was dissolved at 80 degrees C. 0.5g and 0.05g of sodium carbonates were added, and the magnesium oxide was stirred for 30 minutes at 80 degrees C under reduced pressure. 100ml of ethanol was added to this, and it flowed back for 30 minutes. Furthermore, for 15 minutes and after putting at 75 degrees C, precipitation was removed by the decantation, ethanol distilling off of the supernatant was carried out, and it was condensed. This ethanol processing was performed 3 times and 17g of the refining carnauba wax made into the purpose was obtained. The acid number of this refining wax is 1, and was decolorized compared with the raw material. Moreover, the spot equivalent to a pitch was not detected as a result of TLC analysis.
[0011] Rough rice ****** wax 20g whose example 3 acid number is 15 was dissolved at 80 degrees C. 1g added and the calcium oxide was stirred for 30 minutes at 80 degrees C under reduced pressure. 100ml of ethanol was added to this, and it flowed back for 30 minutes. Furthermore, for 15 minutes and after putting at 75 degrees C, precipitation was removed by the decantation, ethanol distilling off of the supernatant was carried out, and it was condensed. This ethanol processing was performed 3 times and 13g of the refining rice ****** waxes made into the purpose was obtained. The acid number of this refining wax is 9, and was decolorized compared with the raw material. TLC spot equivalent to a pitch was not detected.
[0012] The filter cake obtained at the refining process of the sugar which uses example 4 sugarcane as a raw material was dried, it extracted by the 68-degree C hexane, and the rough wax whose acid number is 15 was obtained. After dissolving this rough wax 20g at 80 degrees C, the sodium-hydroxide aqueous solution (0.21g / 10ml) was added, and

it stirred at 80 degrees C. 1g of calcium chlorides was added 2 minutes after, and it stirred at 90 degrees C for 15 minutes to the pan. Subsequently, the bottom of reduced pressure, and after dehydrating at 80 degrees C, 200ml of ethanol was added and it flowed back for 30 minutes. Furthermore, for 15 minutes and after putting at 75 degrees C, precipitation was removed by the decantation, the supernatant was cooled and the produced crystal was carried out the ** exception. This was deliquored at 100 degrees C and 8g of refining waxes was obtained. The acid number of this refining wax is 3, and was decolorized compared with the rough wax of a raw material.

[0013]

[Effect of the Invention] According to this invention, a vegetable rough wax can be decolorized simultaneously with the deoxidation by simple and few operation of a cost-load, and a pitch can also be removed efficiently. Moreover, since the additive with which safety was checked is used, the refining wax obtained by this invention begins food, cosmetics, the drug, etc., and they can be used for it in comfort in various fields.

[Translation done.]